

Application No. 10/589,659
Declaration under 37 C.F.R. 1.132

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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
HORST BINDER, ET AL. : EXAMINER: LEONARD, MICHAEL L
SERIAL NO: 10/589,659 :
FILED: AUGUST 16, 2006 : GROUP ART UNIT: 1796
FOR: METHOD FOR THE PRODUCTION :
OF POLYISOCYANATES COMPRISING
ISOCYANURATE GROUPS AND USE
THEROF

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

Sir:

Now comes Horst Binder, who deposes and states that:

1. I received my Dist. Ing. degree from F.H. Dablen in the year
1980.

2. I have been employed by BASF. CE, the assignee of
the above-identified application, since 1984.

3. I understand the English language or, at least, that the contents of the Declaration
were made clear to me prior to executing the same.

4. The following experiments were carried out by me or under my direct supervision
and control.

5. To illustrate the unexpected benefits of the claimed invention, the effect of an increased amount of free acid in the reaction was evaluated by preparing a series of reactions using different catalysts.

The catalysts were prepared by reaction of tetramethyl ammonium hydroxide with the appropriate acid. The acid was used

- in stoichiometric amounts with regard to the hydroxide ions,
- in a stoichiometric surplus of 10 mol%, and
- in a sub-stoichiometric amount of 10 mol%.

The carboxylic acid (lactic acid) was initially charged in a beaker and the tetrasubstituted ammonium hydroxide, dissolved as a 25% solution in methanol, was added under stirring. On completion of addition of the ammonium hydroxide, the mixture was stirred over night at room temperature. Subsequently, water of reaction formed and other volatile constituents were removed on a rotary evaporator at 90°C and 3 mbar. The residue was degassed and dissolved in 2-ethylhexanol as a 10% solution.

HDI was reacted to the isocyanurate trimer using these catalysts as described in the present specification. The reaction was conducted until a certain NCO value was reached and the reaction was stopped. The reaction mixture was distilled and the color number of the reaction mixture was determined before distillation and after distillation.

For comparative purposes DABCO® TMR was used as a catalyst, as in Comparative Example 1 in the present specification.

6. The results with tetramethylammonium lactate as catalyst are as follows:

	sub-stoichiometric amount of acid	stoichiometric amount of acid	stoichiometric surplus of acid	DABCO® TMR (comparative)
Reaction Time	25 min	35 min	55 min	65 min
NCO content	40.5%	40.5%	41.0%	41.6%
Color number (before distillation)	8 Hz	13 Hz	36 Hz	34 Hz
Color number (after distillation)	31 Hz	54 Hz	111 Hz	80 Hz
Amount of catalyst	202 ppm	204 ppm	230 ppm	265 ppm

7. From the table above, it can easily be seen, that with the catalyst with a 10% stoichiometric surplus of the acid the reaction takes longer and needs more catalyst compared with the catalyst with a stoichiometric amount or a 10% sub-stoichiometric amount. However, the color number is higher and, therefore, not acceptable. In contrast, the catalyst with the stoichiometric and sub-stoichiometric amount of acid provides products with a much better color number.

8. A second series of examples were prepared in which the influence of acids on the reaction of HDI is examined.

For that purpose under nitrogen approx. 9 g HDI were placed into an ampoule, optional 1000 ppm acid and/or 500 ppm catalyst (benzyl trimethylammonium 2-hydroxy-iso-butyrate) was added, and the reaction mixture was heated to 90°C for 60 min. A 2 ml syringe was put into the gas phase of the ampoule in order to compensate pressure, presumably due to carbon dioxide formation.

After that time the reaction was stopped with 2-hydroxyethyl carbamate and cooled down to room temperature. The NCO content and the color number of the reaction mixture was measured.

9. The results of this experiment are as follows:

Example		NCO content (%)	Color number (Hz)	Vapor Compensation (ml)
1	Pure HDI	49.4	11	
2	+ 1000 ppm acetic acid	49.3	8	
3	+ 1000 ppm acetic acid + 500 ppm catalyst	37.6	161	0.8
4	+ 1000 ppm lactic acid	48.9	8	
5	+ 1000 ppm lactic acid + 500 ppm catalyst	36.8	189	2.0
6	+ 500 ppm catalyst	38.5	65	1.0

10. As can be seen from examples 2 and 4 no reaction takes place in the presence of acid. A reaction takes place only in the examples 3,5, and 6, in which catalyst is present. However, in those examples 3 and 5 in which the acid is present when the reaction takes place the color number deteriorates dramatically, regardless of the nature of the acid.

This demonstrates what was pointed out above, that the teaching of Joern et al. is not applicable for the formation of polyisocyanates for coatings: The presence of acids leads to products with a high color number.

11. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false

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statements and the like so made are punishable by fine or imprisonment, or both, under
Section 1001 of Title 18 of the United States Code and that such willful false statements may
jeopardize the validity of this application or any patent issuing thereon.

12. Further deponent saith not.

Horst Binder
Name: Horst Binder

6.8.11
Date